

Krzysztof ROKOSZ, Tadeusz HRYNIEWICZ*, Winfried MALORNY**,
Jan VALICEK***, Lenka CEPOVA****

SEM ANALYSIS OF AISI 316L SS SURFACES AFTER MAGNETOELECTROCHEMICAL
POLISHING MEP TREATMENT IN A TRANSPASSIVE REGION OF POLARISATION
CHARACTERISTICS

SEM ANALÝZA AISI 316L SS POVRCHŮ PO MAGNETOELEKTROCHEMICKÉM LEŠTĚNÍ
MEP OŠETŘENÍ V PASIVNÍ OBLASTI POLARIZAČNÍCH VLASTNOSTÍ

Abstract

The aim of the paper was to carry out the SEM analysis of the Type AISI 316L stainless steel after electropolishing in comparison with the steel surface after magnetoelectropolishing operation. The electrochemical polishing of the AISI 316L SS samples was performed in a transpassive region of polarisation characteristics with the use of a magnetic field induced by a permanent magnet under varying induction (starting from $B = 0$). Following the assumed study method, the effect of magnetoelectropolishing operation on the surface finish was found. The analyses of the SEM and EDX results of AISI 316L samples after MEP obtained in this work show that the magnetic field considerably affects the surface structure and the surface appearance. The studies carried out on AISI 316L SS after MEP under varying conditions allowed to revealing the possible optimal surface

Abstrakt

Cílem práce bylo provést analýzu SEM AISI 316L nerez oceli po elektroleštění ve srovnání s ocelovým povrchem po magnetoelektrolešticí operaci. Elektrochemické leštění vzorků AISI 316L SS byla provedena v pasivní oblasti polarizačních charakteristik s využitím magnetického pole vyvolané permanentními magnety různých indukcí (od $B = 0$). Podle předpokladu, byl zjištěn účinek magnetoelektrického leštění na povrchu. Analýzy SEM a EDX výsledků AISI 316L vzorků po MEP získané v této práci ukazují, že magnetické pole značně ovlivňuje strukturu a vzhled povrchu. Studie prováděné na AISI 316L SS po MEP za různých podmínek odhalila možnosti optimálního povrchu.

1 INTRODUCTION

It has been known that the uniform corrosion of stainless steels is rather low [1]. On the other hand, one of the most critical factors affecting the corrosion behaviour of stainless steels is pitting [1-3]. In the process of electropolishing of stainless steels the avoidance of pitting in the corrosion media is one of the most important tasks and it is possible usually when the process is carried out under a relatively low anodic current density [2-5]. The practice of electropolishing process turns the attention onto the avoidance of metal surface pitting after the operation which is dependent on the electrochemical conditions of treatment, possible usually when the process is carried out on the plateau level [5, 6]. On the other hand, the industrial practice shows that, in view of getting a reasonable and better efficiency, the process of electropolishing is usually carried out in the

* PhD. MSc. Eng., Koszalin University of Technology, Faculty of Mechanical Engineering, Department of Surface Electrochemistry, ul. Raclawicka 15-17, PL 75-620 Koszalin, Poland, tel. (+48) 94 3478 354, e-mail rokosz@tu.koszalin.pl

** Prof. PhD. Eng., Hochschule Neubrandenburg, Brodaer Straße 2, 17033 Neubrandenburg, Germany, tel: (+49) 395 5693 317, e-mail malorny@hs-nb.de

*** Assoc. Prof. Ph.D., MSc. VSB – Technical University of Ostrava, Faculty of Mining and Geology, Institute of Physics, 17. listopadu 15/2172, Ostrava, tel. (+420) 59 732 3232, e-mail jan.valicek@vsb.cz

**** Ph.D., MSc. VSB – Technical University of Ostrava, Faculty of Mechanical Engineering, Department of Machining and Assembly, 17. listopadu 15/2172, Ostrava, tel. (+420) 59 732 4396, e-mail lenka.cepova@vsb.cz

transpassive region of the polarisation curve. Our experiments show that the resultant surface quality may be affected by the magnetic field introduced to the electropolishing system [7]. Extended research studies on magnetoelectro-chemical polishing MEP of stainless steels indicate a significant improvement of numerous surface properties [7-16] in comparison with the results obtained after a conventional electropolishing.

The purpose of the work was to carry out the surface analyses of AISI 316L SS samples after the electrochemical polishing performed in a wide range of current density i and a magnetic field induced by a permanent magnet under varying induction B . The complete statistical programme of investigation was adopted [17, 18] on the basis of the five-level composite rotary plan for three variables: magnetic induction B , current density i , and the pitting potential E_{pit} . The sample surfaces were analyzed using SEM and EDX techniques.

2 METHOD

2.1 Material

The austenitic AISI 316L stainless steel was chosen to study the effect of a magnetic field strength, used during electrolytic polishing, on the final surface quality. The composition of the material used is given in **Table 1** [19]. The samples were prepared in the form of rectangular plates of dimensions 5×30 mm cut of the metal sheet 1 mm thick.

Tab. 1. Chemical Composition of Type 316L Stainless Steel, Vacuum Melted [1]

| Element | Content (wt%) |
|---------|-------------------|
| Cr | 17.38 |
| Ni | 13.78 |
| Mo | 2.76 |
| Mn | 1.84 |
| Si | 0.55 |
| P | Less than 170 ppm |
| S | Less than 10 ppm |
| C | Less than 180 ppm |
| N | Less than 710 ppm |
| Cu | Less than 610 ppm |
| Fe | Balance |

2.2 Set up and parameters

The set up used for the standard electrochemical polishing (EP) is presented in **Fig. 1**. It consists with a potentiostat (A), heating system (B), electrochemical cell (C), computer with the software (D), and a digital multimeter (E).

The developed set up used for the magnetoelectropolishing (MEP) is shown in **Fig. 2**, with the scheme of connections (**A**) and a photograph (**B**) presenting: : a – thermometer, b – anode, c – cathode, d – electrochemical cell, e – heater, and f – neodymium magnet. The main elements of the set up involved also a mixing device, a dc power supply, the electrodes and connecting wiring, a potentiostat, and a controller. The studies were carried out in a broad range of changes of the current density (up to 1000 A/dm²), potential up to 24 V vs. a reference electrode, and the electrolyte temperature of 20 to 100 °C, at the temperature control equalled ±1 °C. For the studies a proprietary sulphuric/orthophosphoric acids mixture electrolyte was used. To eliminate any possible change in the electrolyte composition due to the increased temperatures, no water was used for the electrolyte in the study. For each run the electrolytic cell, made of glass, contained up to 500 cm³ of electrolyte.

The samples were prepared in the Division of Electrochemistry, Faculty of Mechanical Engineering, Koszalin University of Technology, degreased in acetone, and then electropolished in accordance to the statistical plan presented in **Fig. 3**.



Fig. 1 Set up used for electrochemical polishing ($B = 0$ mT), A – potentiostat, B – heating system, C – electrochemical cell, D – computer with software, E – digital multimeter

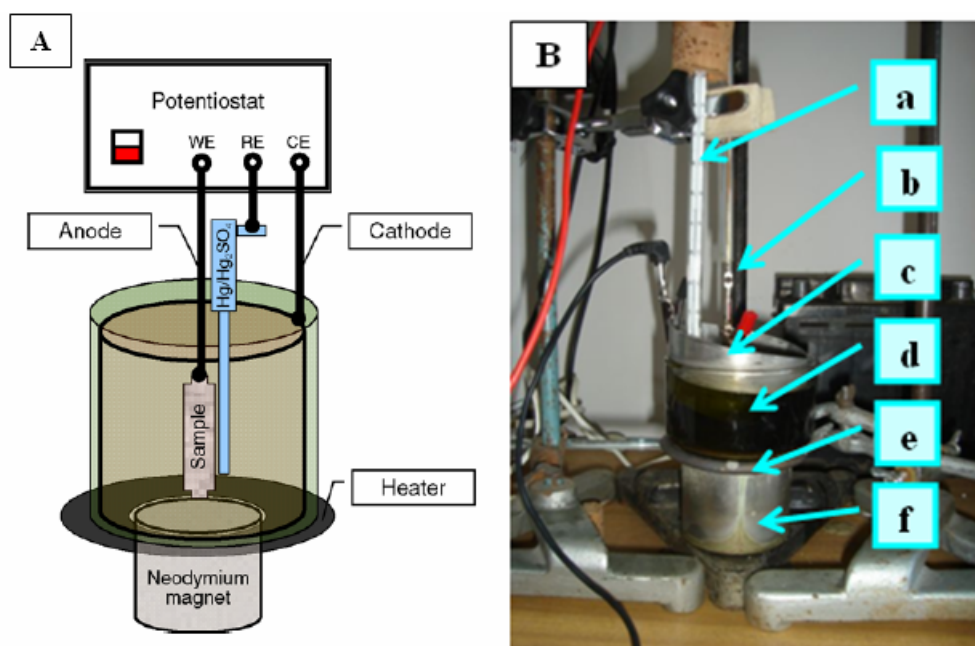


Fig. 2 Set up used for magnetoelectropolishing MEP: A – scheme of connections, B – photo with details: (a – thermometer, b - anode, c - cathode, d - electrochemical cell, e - heater, f - neodymium magnet)

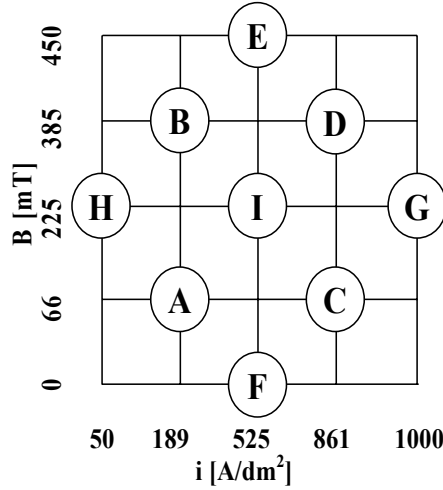


Fig. 3 Statistical plan of experiment: (a) $B = 66$ mT, $i = 189$ A/dm², (b) $B = 384$ mT, $i = 189$ A/dm², (c) $B = 66$ mT, $i = 861$ A/dm², (d) $B = 384$ mT, $i = 861$ A/dm², (e) $B = 450$ mT, $i = 525$ A/dm², (f) $B = 0$ mT, $i = 525$ A/dm², (g) $B = 225$ mT, $i = 1000$ A/dm², (h) $B = 225$ mT, $i = 50$ A/dm², (i) $B = 225$ mT, $i = 525$ A/dm²

The complete statistical programme of investigation was adopted [17, 18] on the basis of the five-level compositive rotary plan for three variables: magnetic induction B , current density i , and the pitting potential E_{pit} . The sample surfaces were analysed using SEM and EDX techniques.

3 RESULTS AND DISCUSSION

The basic analysis of the samples after MEP was carried out using SEM and EDX. The EDX results of the electropolished AISI 316L SS sample surface are presented in Fig. 4.

In the picture (Fig. 4), two dominating elements constituting the AISI 316L SS are visible, which are iron and chromium. Two additional elements, nickel and molybdenum, may be also noticed.

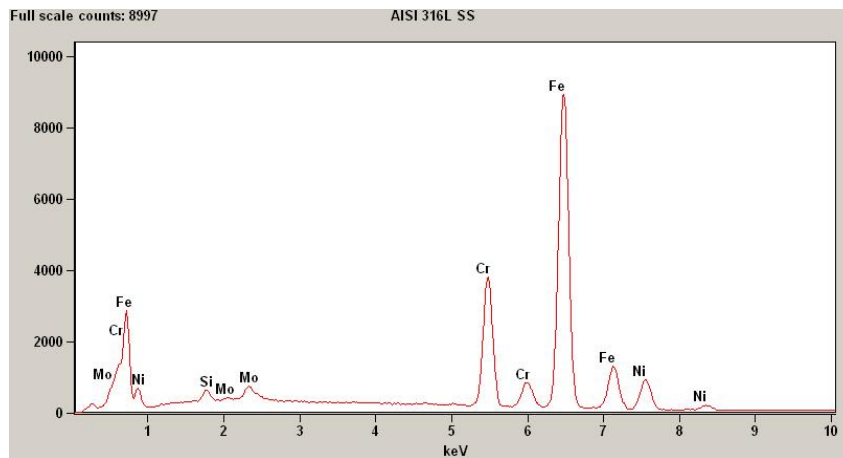
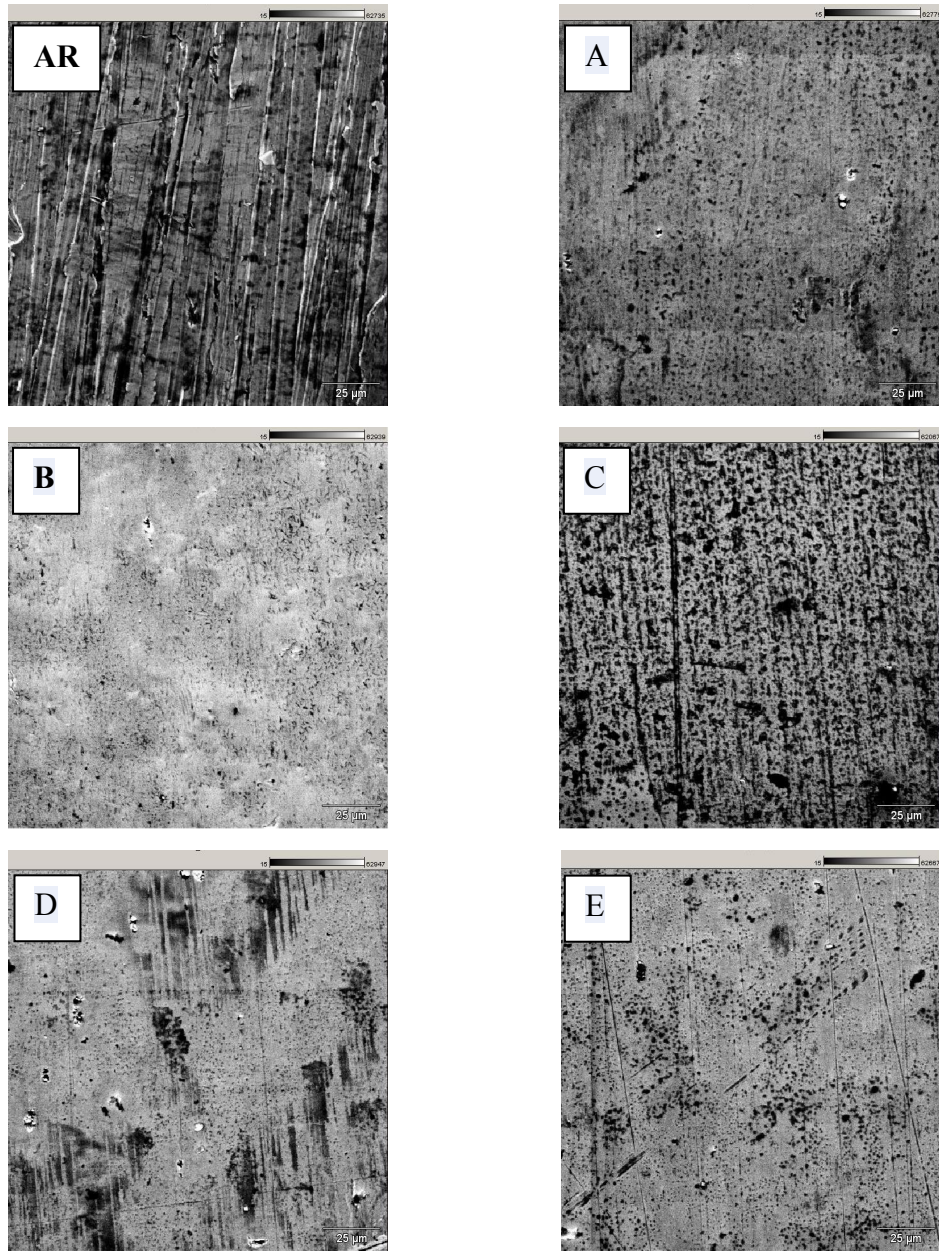


Fig. 4 EDX results of electropolished AISI 316L SS sample

In Fig. 5 the SEM results of the treated samples of AISI 316L SS are given. Fig. 5 AR presents the sample in the initial state, received after abrasive polishing with the grain size up to 1000. In the next pictures (Fig. 5 A through I), the SEM images after electropolishing under different conditions of current density and the magnetic induction, in accordance to the plan of experiment,

have been presented. Each of the surface image given in Fig. 5 reveals some characteristic features, differing from case to case. The SEM image of the steel sample after MEP at $B = 66$ mT, and $i = 189$ A/dm² with very few traces of discontinuities on the surface is presented in Fig. 5 A. The next SEM image, presented in Fig. 5 B, after MEP at $B = 66$ mT, $i = 861$ A/dm², appeared to be the best of all studied cases.



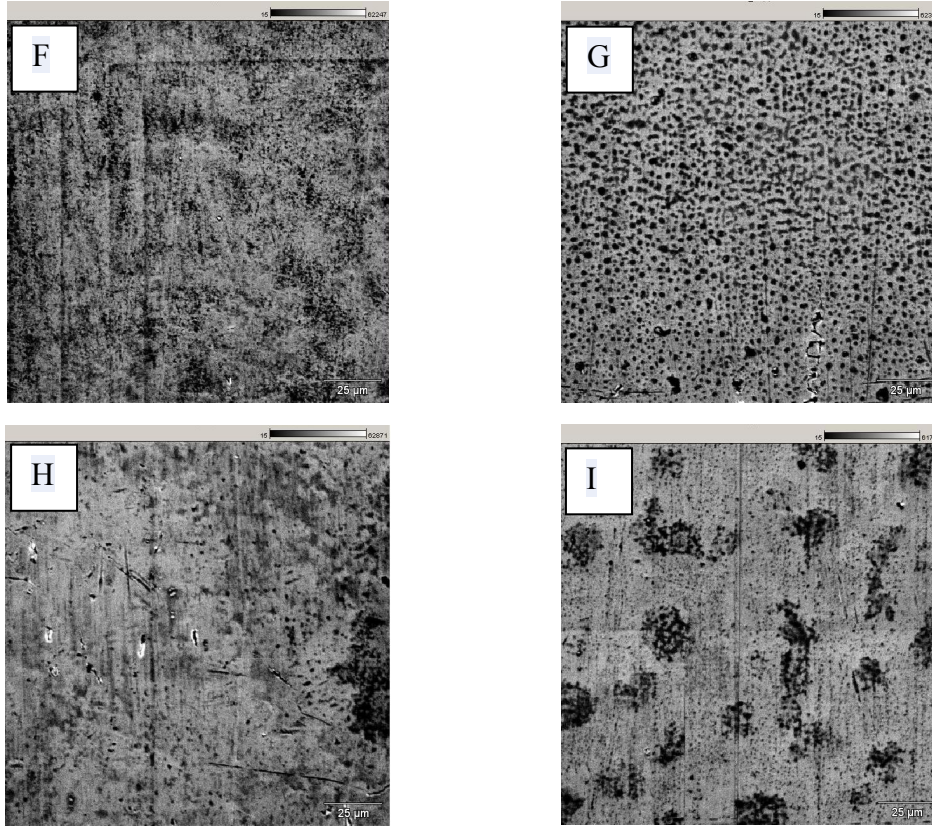


Fig. 5 SEM results of the treated samples of AISI 316L SS in different conditions: AR – as received after abrasive polishing, and after electropolishing at: (A) $B = 66$ mT, $i = 189$ A/dm², (B) $B = 384$ mT, $i = 189$ A/dm², (C) $B = 66$ mT, $i = 861$ A/dm², (D) $B = 384$ mT, $i = 861$ A/dm², (E) $B = 450$ mT, $i = 525$ A/dm², (F) $B = 0$ mT, $i = 525$ A/dm², (G) $B = 225$ mT, $i = 1000$ A/dm², (H) $B = 225$ mT, $i = 50$ A/dm², (I) $B = 225$ mT, $i = 525$ A/dm²

The following SEM images, given in Fig. 5 C through I, were performed after MEP at the conditions: (C) $B = 66$ mT, $i = 861$ A/dm²; (D) $B = 384$ mT, $i = 861$ A/dm²; (E) $B = 450$ mT, $i = 525$ A/dm²; (F) $B = 0$ mT, $i = 525$ A/dm²; (G) $B = 225$ mT, $i = 1000$ A/dm²; (H) $B = 225$ mT, $i = 50$ A/dm²; and (I) $B = 225$ mT, $i = 525$ A/dm². All they are characteristic with some traces of irregularities and pits on the surface. Such results indicate on the necessity to choose proper MEP conditions, concerning the final surface quality.

In **Fig. 6** a, b two enlarged images of the best surfaces, with fine or very fine traces of pits, are presented. They result from the assumed conditions of electrochemical polishing, above the plateau of polarisation curve, though one of the reasons for the occurrence of pits and/or discontinuities may be the inclusions present in the surface layer of the treated material.

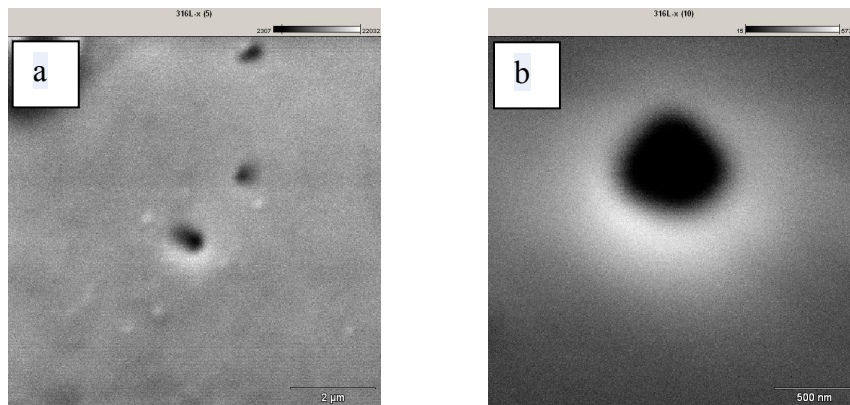


Fig. 6 Typical SEM results of polished samples of AISI 316L SS after MEP at $B \approx 384$ mT and current densities between 189 A/dm^2 and 525 A/dm^2

4 CONCLUSION

The following conclusions may be formulated after the investigation with the results presented above:

- The increasing magnetic field applied in the electropolishing system results in a rotation of the solution in electrochemical cell, thus affecting negatively the MEP process at extreme conditions
- The magnetic field introduced during electropolishing has a big influence on the pitting corrosion resistance of stainless steels
- The EDX study results show the most important elements of the AISI 316L stainless steel (see Fig. 4), i.e. compounds of iron and chromium with the addition of nickel and molybdenum, being in agreement with the chemical composition
- The best electropolished surfaces were found on samples A and B (see Fig. 5 A,B) obtained after MEP in the magnetic field of $B \approx 384$ mT at the current density $i = 189 \text{ A/dm}^2$
- MEP at higher current densities, above 525 A/dm^2 reveals the occurrence of pits on the treated surface
- MEP at the current densities between 189 and 525 A/dm^2 has revealed quite well electropolished surfaces with the areas of fine pits (see Fig. 6).

REFERENCES

- [1] HRYNIEWICZ T., *Electrochemistry for Surface Engineering (Elektrochemia dla Inżynierii Powierzchni)*, Monograph ed. by T. Hryniewicz, Politechnika Koszalińska, Koszalin, 2005 (386 pages)
- [2] PISTORIOUS P.C., and BURSTEIN G.T., *Metastable Pitting Corrosion of Stainless Steel and the Transition to Stability*, Phil.Trans.R.Soc., Physical, Mathematical and Engineering Sciences, 1992, 341(1662), 531-559
- [3] HRYNIEWICZ T., MULLER R.H., and TOBIAS C.W., *A Study of Electropolishing of Ferrous Alloys Using Rotating Disk Electrodes*, Work prep. for the US Dept. of Energy under Contract no. W-7405 ENG-48, Materials And Molecular Research Division, Lawrence Berkeley Laboratory, University of California, Berkeley CA, June 1981, (LBL-12879 and UC-25), (122 pages).
- [4] HRYNIEWICZ T., *Concept of microsmoothing in the electropolishing process*, Surface & Coatings Technology, 1994, 64(2), 75-80.
- [5] DETTNER P., *Electrolytic and Chemical Polishing of Metals*, Copyright 1987, Ordentlich Publishers Second Printing, May 1988, Express-SDAR Ltd., Needar Press, Tel Aviv, 340 p.

- [6] TEGART W.J. McG., *The Electrolytic and Chemical Polishing of Metals in Research and Industry*, 2nd ed. Pergamon Press, New York, 1959
- [7] HRYNIEWICZ T., ROKICKI R., ROKOSZ K., *Magneto electropolishing Process Improves Characteristics of Finished Metal Surfaces*, Metal Finishing, December, 2006, Vol. 104, Nr 12, 26-33
- [8] HRYNIEWICZ T., ROKICKI R., ROKOSZ K., *Metal Surface Modification by Magneto electropolishing*, Proc. of METAL2007, 22-24 May, 2007, Hradec nad Moravici, Czech Republic, Symp. D, Surf.Engng., D2, 1-8
- [9] HRYNIEWICZ T., ROKICKI R., ROKOSZ K., *Magneto electropolishing for metal surface modification*, Transactions of the Institute of Metal Finishing, Vol. 85, No 6/November, 2007, 325-332
- [10] HRYNIEWICZ T., ROKICKI R. *On the surface modification of stainless steels*, Proc. of 15th Annual Intl.Confer. on Composites/Nano-Engineering, ICCE-15, 15-21 July, 2007, Haikou, Hainan Island, China, ed. by David Hui, pp. 345-346
- [11] HRYNIEWICZ T., ROKICKI R., ROKOSZ K., *Surface characterization of AISI 316L biomaterials obtained by electropolishing in a magnetic field*, Surface & Coatings Technology, Available online 7 August 2007, 1 February 2008, Vol. 2002, issue 9, 1668-1673
- [12] HRYNIEWICZ T., ROKICKI R., ROKOSZ K., *Corrosion Characteristics of Medical Grade AISI 316L Stainless Steel Surface after Electropolishing in a Magnetic Field*, The Journal of Corrosion Science and Engineering (Bob Cottis), Vol. 10, Preprint 45, subm. 3 August, 2007, 1-10
- [13] HRYNIEWICZ T., ROKICKI R., ROKOSZ K., *Improving of corrosion characteristics of austenitic stainless steels surface by magneto electropolishing*, Proc. of METAL'2008, Hradec nad Moravici, Czech Republic, 13-15 May, 2008
- [14] HRYNIEWICZ T., ROKOSZ K., ROKICKI R., *Comparison of Corrosion Behaviour of Austenitic Steels after Standard Electropolishing and Magneto electropolishing*, Proc. of EUROCORR 2008, Edinburgh, 7-11 September, 2008, Poster Session.
- [15] HRYNIEWICZ T., ROKOSZ K., ROKICKI R., *Electrochemical and XPS Studies of AISI 316L Stainless Steel after Electropolishing in a Magnetic Field*, Corrosion Science, 50(9) (2008) 2676-2681
- [16] HRYNIEWICZ T., ROKOSZ K., FILIPPI M., *Biomaterial Studies on AISI 316L Stainless Steel after Magneto electropolishing*, Materials, 2009, 2(1), 129-145
- [17] KUKIELKA L., *Fundamentals of engineering studies (Podstawy badań inżynierskich)*, Koszalin, Politechnika Koszalińska, PWN, Warszawa, 2002
- [18] KUKIELKA S., *Experiment Planner 1.0. – A software for experimental planning*, MSc Thesis, Koszalin, Politechnika Koszalińska, 2003
- [19] *Metals Handbook*, 10th Edition, Vol. 1, *Properties and Selection. Irons, Steels and High-Performance Alloys*, ASM International, Materials Park, OH 44073, 1990